Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2-Dimethyl-5-[(4-*p*-tolylthiazol-2-ylamino)-methylene]-1,3-dioxane-4,6-dione

Luiz Everson da Silva,[a,b] Antonio Carlos Joussef,[a] Sabine Foro[b] and Boris Schmidt[b]*

[a]Departamento de Química–UFSC, 88040-900 Florianópolis, SC, Brazil, and [b]Clemens Schöpf-Institut für Organische Chemie und Biochemie, Technische Universität Darmstadt, Petersenstrasse 22, D-64287 Darmstadt, Germany

Correspondence e-mail: foro@tu-darmstadt.de

Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.107
Data-to-parameter ratio = 12.8

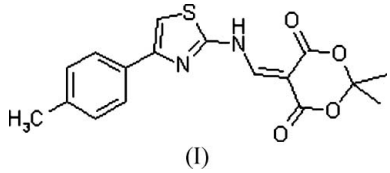
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 June 2006
Accepted 29 June 2006

In the title compound, $C_{17}H_{16}N_2O_4S$, the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation. The amino H atom has an intramolecular contact to a carbonyl O atom, forming a six-membered ring.

Comment

Many heterocyclic derivatives of Meldrum's acid (2,2-dimethyl-1,3-dioxane-4,6-dione) have been prepared and well studied from the synthetic and structural points of view (Chen, 1991), whereas their biological properties have scarcely been investigated. On the other hand, thiazoles and their derivatives are found to be associated with various biological activities such antibacterial and anti-inflammatory (Holla *et al.*, 2003). Moreover, recently, Farghaly *et al.* (2006) described the synthesis of azoles with potential antiviral activity. Thus, as part of our screening program to investigate the antiviral activity of 5-arylaminemethylene Meldrum's acid derivatives (da Silva *et al.*, 2005*a*,*b*, 2006, 2006*a*,*b*), we have performed an investigation of the crystal structure of the title compound, (I).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation with C14 in the flap position. The C12—C11—N2—C1 torsion angle of −176.7 (2)° and the C11—N2 and C11—C12 distances (Table 1) indicate delocalization. The delocalization of the N atom lone pair into the Meldrum's acid

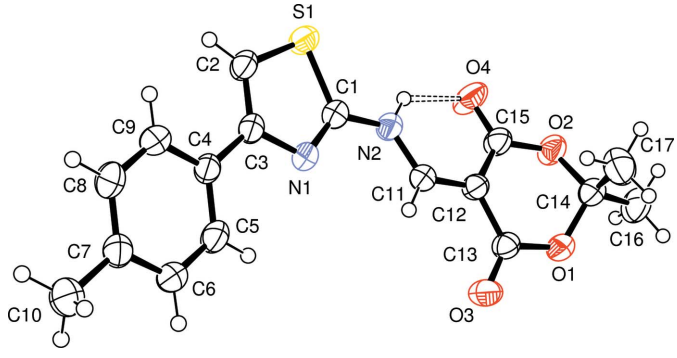


Figure 1
The molecular structure of (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is shown as dashed lines.

ring may be favoured in the direction of one of the two available carbonyl groups C13═O3 and C15═O4 (Blake *et al.*, 2003). The thiazole ring and the benzene ring are slightly twisted with respect to each other, with a dihedral angle of 9.8 (1)° between the mean planes. The H atom of the NH group makes one intramolecular hydrogen bond to atom O4 (Table 2), forming a six-membered ring.

Experimental

The title compound was prepared according to the literature procedure (Cassis *et al.*, 1985) and was recrystallized from methanol (m.p. 490 K).

Crystal data

$C_{17}H_{16}N_2O_4S$
M_r = 344.38
Triclinic, $P\overline{1}$
a = 7.7951 (8) Å
b = 9.5345 (9) Å
c = 11.805 (1) Å
α = 71.034 (9)°
β = 78.564 (9)°
γ = 76.671 (9)°
V = 800.11 (13) Å^3
Z = 2
D_x = 1.429 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 2.02 mm^{-1}
T = 299 (2) K
Prism, light brown
0.20 × 0.11 × 0.05 mm

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.814, T_{max} = 0.998
(expected range = 0.738–0.904)
5625 measured reflections
2854 independent reflections
2014 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 66.9°
3 standard reflections
frequency: 120 min
intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.107
S = 1.01
2854 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.1684P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected bond lengths (Å).

C11—N2	1.336 (3)	C11—C12	1.364 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O4	0.90 (2)	2.00 (3)	2.699 (3)	134 (2)

H2*N* was found in a difference map and was refined isotropically. The carbon-bound H atoms were including in the riding-model approximation, with C—H = 0.93 (aromatic) and 0.96 Å (methyl). Isotropic displacement parameters for all H atoms were set equal to $1.2U_{eq}$(parent atom).

Data collection: *CAD-4 Software* (Nonius, 1996); cell refinement: *CAD-4 Software*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank Professor Dr Hartmut Fuess, FG Strukturforschung, FB Material- und Geowissenschaften, Technische Universität Darmstadt, for diffractometer time.

References

Blake, A. J., McNab, H. & Withell, K. (2003). *Acta Cryst.* E**59**, o841–o842.
Cassis, R., Tapia, R. & Valderrama, J. A. (1985). *Synth. Commun.* **15**, 125–129.
Chen, B. C. (1991). *Heterocycles*, **32**, 529–597.
Farghaly, A.-R. & El-Kashef, H. (2006). *Arkivoc*, **XI**, 76–90.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Holla, B. S., Malini, K. V., Rao, B. S., Sarojini, B. K. & Kumari, N. S. (2003). *Eur. J. Med. Chem.* **38**, 313–318.
Nonius (1996). *CAD-4-PC Software.* Version 1.2. Nonius, Delft, The Netherlands.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Silva, L. E. da, Joussef, A. C., Andrighetti-Fröhner, C. R., Nunes, R. J. & Bortoluzzi, A. J. (2006). *Acta Cryst.* E**62**, o1062–o1063.
Silva, L. E. da, Joussef, A. C., Foro, S. & Schmidt, B. (2006*a*). *Acta Cryst.* E**62**, o742–o743.
Silva, L. E. da, Joussef, A. C., Foro, S. & Schmidt, B. (2006*b*). *Acta Cryst.* E**62**, o1722–o1723.
Silva, L. E., Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*a*). *Acta Cryst.* E**61**, o4121–o4122.
Silva, L. E., Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*b*). *Acta Cryst.* E**61**, o4252–o4253.
Stoe & Cie (1987). *REDU4*. Version 6.2c. Stoe & Cie GmbH, Darmstadt, Germany.